

FIG. 1A

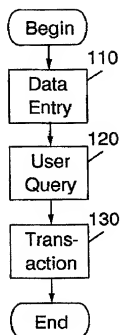


FIG. 1B

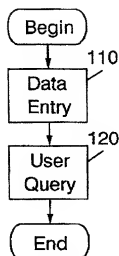


FIG. 1C

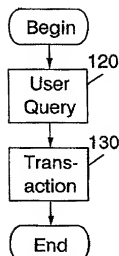


FIG. 1D

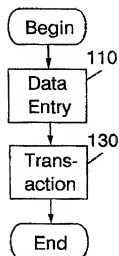


FIG. 1E

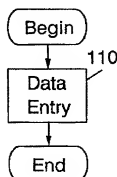


FIG. 1F

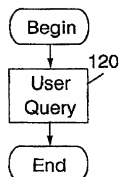
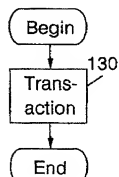


FIG. 1G



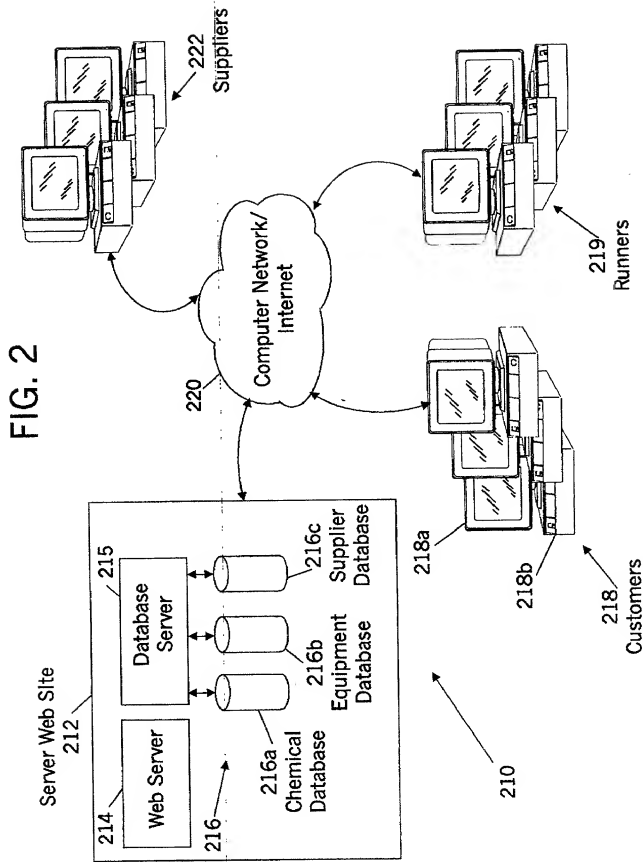
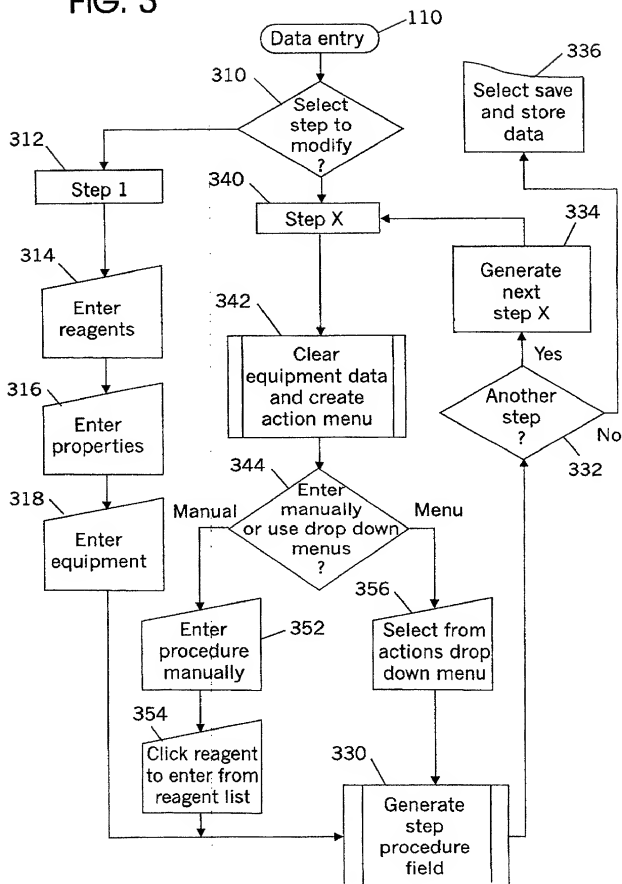


FIG. 3



Synthetic Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents:

	CAS	Name	Weight	Amount (g)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				

Starting Flask:

Equipped with:

Procedure:

Step:

Reaction type/keywords (Separated by semicolon):

FIG. 4

[illegible]

FIG. 5

Synthematrix Protocol Manager ver. 0.90b

Name:

CAS:

Readings

	CAS	Name	Weight	Formula	Density	BP	FP	MP	Vapor Pressure	Comments	Beilstein	Other Names
1	<input type="text" value="101-01-1"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>

Procedure

Previous:

Save

Cancel

Exit

Go To Procedure

New

Save

Update

FIG. 6

Synthmaster Protocol Manager ver. 0.90b

Name: CAS: Formula: Weight:

Reagents ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Actions: <input type="text"/>
2	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Qualifier: <input type="text"/>
3	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Action: <input type="text"/>
4	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Reagent: <input type="text"/>
5	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	Time: <input type="text"/>
6	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="button" value="Reset"/> <input type="button" value="Add To Procedure"/>
7	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
8	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
9	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	
10	<input type="text"/>	<input type="text"/>	<input type="text"/>	<input type="text"/>	

Procedure

Step 2

Yield: Density:

BP: IP:

MP: Vapor Pr:

Ballston: Other Names:

FIG. 7

09722229.012901

[illegible]

FIG. 8

SynthMatrix Protocol Manager ver. 0.90q

File Apparatus Atmospheres Operations

Name: CAS: Formula: Weight:

6-(allylamino)-5-amino-4-chloropyrimidine R00-02-3 C7H9N4Cl 184.63

Reagents benzene **REORDER**

CAS	Name	Weight	Amount (g)
R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50
107-11-9	2-Propanol-1-amine	57.09	100
64-17-5	Ethanol	46.07	100
71-43-2	Benzene	78.11	3146.4

Starting Flask

Round bottom 3-neck flask

Equipped with:

Procedure

Previous **Next**

Step 1

Into a round bottom 3-neck flask was added <GND> grams (<MND> mol) of 5-amino-4,6-dichloropyrimidine

Info **New**

Properties **Save**

Reference **Extra Equip.** **Waste**

FIG. 10

Synthetix Protocol Manager ver. 0.90a

File Apparatism Automate Operations

Name: CAS: Formula: Weight:

6-(allylamino)-5-amino-4-chloropyrimidine R00-02-3 C7H9N4Cl 184.63

Reagents benzene ☐ Enter procedure by hand

CAS	Name	Weight	Amount (g)
R00-02-4	5-amino-3,5-dichloropyrimidine	163.99	50
107-11-8	2-Propenylamine	57.09	100
64-17-5	Ethanol	46.07	100
71-43-2	Benzene	78.11	3148.4

Action:

Qualities:

Action:

Reagent:

Time:

Procedure

Step 7

FIG. 11

09772229.012004

Synthetic Protocol Manager ver. 0.90q
 File Apparatus Atmospheric Operations

Name: CAS: Formula: Weight:

8-(ethylamino)-5-amino-4-chloropyrimidine R00-02-3 C7H9N4Cl 184.63

Reagents: benzene Lookup Enter procedure by hand

CAS	Name	Weight	Amount (g)	Action	Qualifier	Repeat	Time
R00-02-4	5-amino-4-chloropyrimidine	163.99	50	To the flask			
107-11-9	2-Propen-1-amine	57.09	100		was		
84-17-5	Ethanol	46.07	100				
71-43-2	Benzene	78.11	3146.4	added		Page 2 of 2	

Reset Add to Procedure

Procedure

Previous Next

Step 2

To the flask was added «GN1» grams («MN1» mol) of 2-Propen-1-amine and «GN2» grams («MN2» mol) of Ethanol

Info New Properties Save Reference Extra Copy Update

FIG. 12

Synthesmate Protocol Manager ver. 0.90q
 File | Apparatus | Atmospheres | Operations

Name: CAS: Formula: Weight:

6-(allylamino)-5-amino-4-chloropyrimidine R00-02-3 C7H9N4Cl 164.63

Reagents: benzene [Lookup] ☐ Enter procedure by hand

	CAS	Name	Weight	Amount (g)	
1	R00-02-4	5-amino-4,6-dichloropyrimidine	163.99	50	Action: []
2	107-11-9	2-Propen-1-amine	57.09	100	Qualifier: []
3	94-17-5	Ethanol	46.07	100	
4	71-43-2	Benzene	78.11	3146.4	Action: []
5					Reagent: []
6					
7					Time: []
8					
9					
10					

[Reset] [Add To Procedure]

Procedure

[Previous] [Next]

Step 2

To the flask was added <QN1> grams (<MN1> mol) of 2-Propen-1-amine and <QN2> grams (<MN2> mol) of Ethanol

[Info] [New] [Properties] [Save] [References] [Extra Setup] [Delete]

FIG. 13

106210*622260

Synhematix Protocol Manager ver. 0.90b

Name: CAS Formula Weight:

Reagents: ☐ Enter procedure by hand.

	CAS	Name	Weight	Amount (g)	Action
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

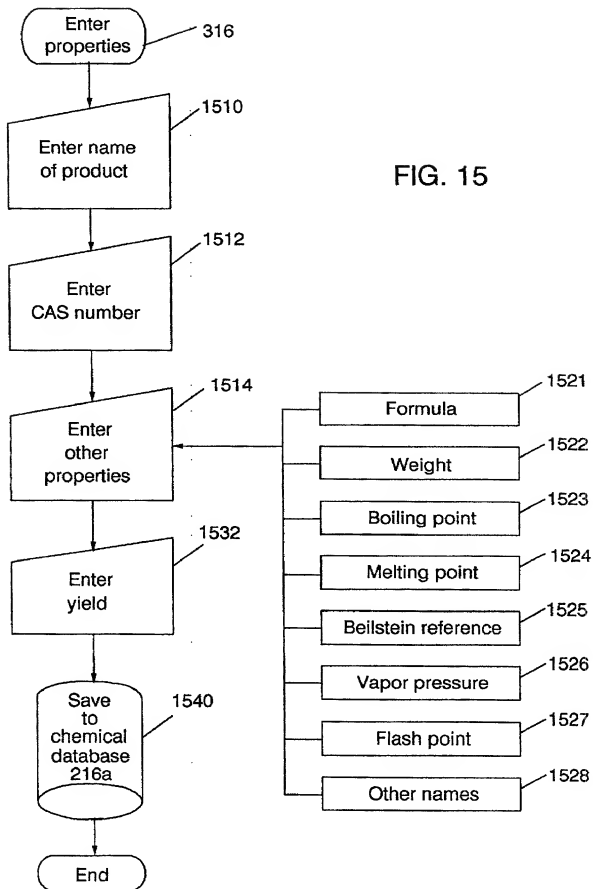
Qualifier: Action: Reagent: Time:

Procedure:

Step 2

Reaction: type keywords (separated by semicolon)

FIG. 14



File: Apparatus: Atmosphere: Operations

Name: CAS: Formula: Weight:

6-(silylamino)-5-amino-4-chloropyrimidine R00-02-3 C7H8N4Cl 184.63

Reagents: benzene ☐ Enter procedure by hand

CAS	Name	Weight	Amount (g)	Action
1 R00-02-4	5-amino-4-chloropyrimidine	163.99	50	<input type="button" value="Action"/>
2 107-11-9	2-Propanol-amine	57.09	100	<input type="button" value="Action"/>

6-

Author: Thomas et al. Journal: acid chemistry

Year: 1968 Vol: 1

Page: 22

Procedure

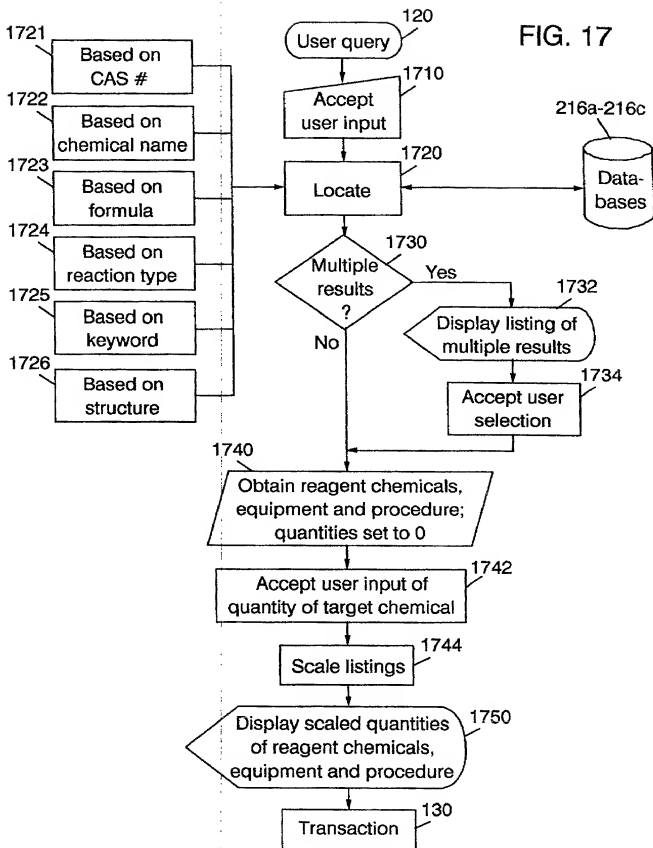
Step 6

The extracts are then evaporated to dryness and can be recrystallized from petroleum ether to give a pure product

FIG. 16

0972229.012504

FIG. 17




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Document Date

FIG. 18

Synthematic Search Results - Netscape

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Location: <http://63.113.160.203/reviews/QueryProjectName=brmo> What's New?

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Chemical Procedures
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Glassware
Stock and custom glassware

Publications
Access scientific papers published online with Synthematic

Advanced
Enter an advanced site-wide search query

Synthematic: Search Results

Number of matches: 7

alpha-bromo-p-toluene boronic acid

n-Butyl-2-bromopropionate

(6-Bromo-2,2-diphenylbenzo[1,3]dioxol-5-yl)methanol

(6-Bromo-2,2-dimethylbenzo[1,3]dioxol-5-yl)methanol

6-bromo-2,2-diphenylbenzo[1,3]dioxole-5-carbaldehyde

6-bromo-3,4-((diphenylmethylene)dioxy)-benzaldehyde ethanedithyl acetal

1-Benzhydryl-3-bromooxetidine

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FIG. 19

2010

2060

2020

2030

2040

2050

Synthetic Compound Procedure - Microsoft Internet Explorer

Address http://63.118.159.203/servelets/ShowChembasePage-16

Links: Home, Chemical Abstracts, Internet Search, Windows Update, RealPlayer, Firefox, Yahoo

n-Butyl-2-bromopropionate (208.08) C7H13BrO3

BrC[C@H](C)C(=O)O + CCCCO → BrC[C@H](C)C(=O)OCCCC + H2O

2-Bromopropionic Acid C₃H₅BrO₂
 Exact Mass 215.96
 Mol Wt. 155.98
 C 21.05 B 12.12
 Br 54.22 O 30.92

1-Bromol C₃H₇Br
 Exact Mass 149.97
 Mol Wt. 149.97
 C 54.82 B 13.60 O 21.59

n-Butyl-2-bromopropionate C₇H₁₃BrO₃
 Exact Mass 208.01
 Mol Wt. 208.01
 C 42.21 H 4.22 Br 30.22 O 15.38

Water Mol Weight mol Calculated

Chemicals needed

Chemical	Formula Weight	Stoichiometry	Moles Needed	Grams Needed
2-Bromopropionic acid	152.96	1	0.0000	0.0000
n-butanol	74.12	1	0.0000	0.0000
Hexane	86.18	1	0.0000	0.0000
Dowex 50WA-200	3.68	1	0.0000	0.0000
Water	18	1	0.0000	0.0000

Equipment needed

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean-Stark Trap
1	Condenser, Allihn, Drop tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Collector
1	500 ml round bottom flask
2	Vacuo
3	Tubing (ft.)

Procedure

1. Into a 3 Neck Flask equipped with an overhead stirrer, Dean stark collector, condenser, and heating mantle was placed g (mol) of 2-Bromopropionic acid
2. To the 2-Bromopropionic acid was added g (mol) of n-butanol.
3. To the 2-Bromopropionic acid and n-butanol was added g (mol) of Hexane.
4. To the flask is added g of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 3.68 grains/mol.
5. Two vacuo are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
6. The reaction is monitored for water generation. After collection g (mol) of water the reaction is complete.
7. Hexane is then removed by continuous drawing of the dean stark collector.
8. The reaction is then cooled to room temperature and filtered through a fluted glass funnel to remove the Dowex resin.
9. The crude n-butyl-2-bromopropionate is then purified via vacuum distillation. (The vacuum distillation procedure is below)

Vacuum distillation

- The product is placed into a 3L round bottom and connected to the pilot scale distillation head equipped with a vacuum column. The material is heated in a 3L mantle set to 45-50% power with a vacuo.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 5 hours.

References

FIG. 20

Systematic Compound Procedure - Microsoft Internet Explorer

Address: 71d-16kmd-126MND-1835.764MNI-126GN1-8893.446MNI-126GN2-1034.164MNI-126GN3-44.164MNI-126GN4-216

Links: 126MNI-126GN1-8893.446MNI-126GN2-1034.164MNI-126GN3-44.164MNI-126GN4-216

2010

2-Bromopropionic Acid + 1-Propanol → n-Butyl 2-bromopropionate + H₂O

2-Bromopropionic Acid: C₃H₅BrO₂, Exact Mass: 151.95, Mol Wt: 152.96, C: 23.85, H: 1.29, Br: 55.20, O: 30.56

1-Propanol: C₃H₈O, Exact Mass: 74.07, Mol Wt: 74.12, C: 64.62, H: 11.00, O: 11.59

n-Butyl 2-bromopropionate: C₇H₁₃BrO₂, Exact Mass: 208.01, Mol Wt: 209.08, C: 41.21, H: 6.27, Br: 39.22, O: 15.30

2060

Enter Mol Scale: 12 mol

2020

Chemicals needed

Chemical	Formula	Weight	Moles	Grams
2-Bromopropionic acid	C ₃ H ₅ BrO ₂	152.96	1	152.96
n-Propanol	C ₃ H ₈ O	74.12	1	74.12
Hexane	C ₆ H ₁₄	86.18	1	86.18
Dowex 50W4-200		3.68	1	3.68
Water	H ₂ O	18	1	18

2030

Equipment needed

Qty	Equipment
1	Round bottom 3-neck flask
1	Overhead stirrer
1	Dean-Stark Trap
1	Condenser, Allihn, Drop tip
1	Heating mantle
1	Recirculating Chiller
1	Heavy duty distillation head
1	Fraction Collector
4	500 ml round bottom flask
2	Vacuum
3	Tubing (ft.)

2040

Procedure

- Into a 3 Neck Flask equipped with an overhead stirrer, Dean-Stark collector, condenser, and heating mantle was placed 1835.7600 grams (12.0000 moles) of 2-Bromopropionic acid.
- To the 2-Bromopropionic acid was added 889.4401 grams (12.0000 moles) of 1-Propanol.
- To the 2-Bromopropionic acid/propanol mix was added 1034.1600 grams (12.0000 moles) of Hexane.
- To the flask is added 3.68 grams of Dowex resin to facilitate the esterification. The resin is previously dried and has an activity of 5-6 gm/cc.
- Two vacuums are attached to the heating mantle and a setting of 50% power is used for heating to rapid reflux.
- The reaction is monitored for water generation. After collecting 216.0000 grams (12.0000 moles) of water the reaction is complete.
- Hexane is then removed by continuous draining of the dean-stark collector.
- The reaction is then cooled to room temperature and filtered through a fritted glass funnel to remove the Dowex resin.
- The crude n-butyl 2-bromopropionate is then purified via vacuum distillation. [The vacuum distillation procedure is below]

Vacuum distillation

- The product is placed into a 5L round bottom and connected to the jacketed distillation head equipped with a Vigreux column. The material is heated in a 5L mantle set to 45-50% power with a vacuum.
- The product is collected at a head temp of 40-45°C. The material rapidly condenses once the apparatus is heated up. Total time for distillation is approx. 3 hours.

2050

References

FIG. 21

FIG. 22

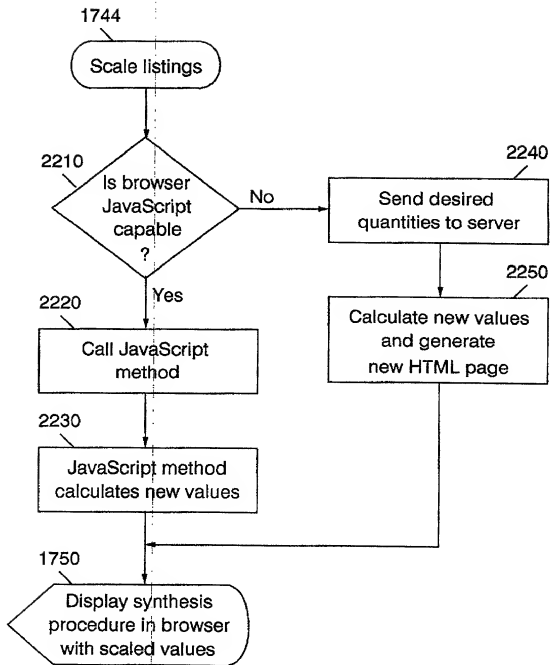


FIG. 23

